IS 3925 (1980): Eugenol [PCD 18: Natural and Synthetic Fragrance Materials]

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SPECIFICATION FOR EUGENOL

(First Revision)

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AMENDMENT NO. 1 AUGUST 1992
TO
IS 3925 : 1980 SPECIFICATION FOR EUGENOL

(First Revision)

(*Page* 3, *clause* **0.3**, *line* 3) —Delete the words 'for guidance'.

(*Page* 3, *clause* **0.3**) — Insert the following new clause after **0.3**:

'**0.3.1** Although in accordance with the general decision of the Committee, GLC method is to be preferred to wet method in case of aromatic chemicals, for eugenol, both the methods, gas charomatography, as well as wet method of determination of phenol are being retained because in a material such as eugenol, there is a possibility of presence of naturally occurring polymeric material or high boiling adulterants which cannot be resolved or detected by GLC. On the other hand there is a possibility of addition of other phenols as adulterants in eugenol which cannot be detected by wet method. Therefore both the methods are being retained in this standard as they are essential and complimentary to each other.'

(PCD 18)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR EUGENOL

(First Revision)

0. FOREWORD

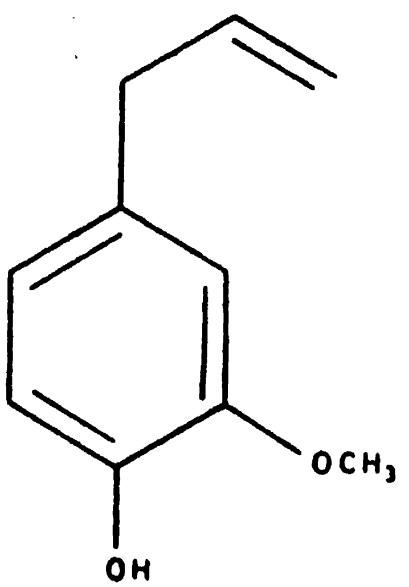
0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 20 June 1980, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1966. The Sectional Committee responsible for the preparation of this standard felt that it should be revised in view of trade practices in perfumery industry and to suit the material currently being manufactured and sold in the country.

0.3 In the present revision, gas chromatographic method for determination of purity of compound which is progressively used in the country, has been included for guidance.

0.4 Eugenol ($C_{10}H_{12}O_2$) is the main constituent of oil of cloves, oil of *Ocimum gratissimum* and oil of cinnamon leaf from *Cinnamomum zeylanicum* (*TEJPAT* leaf oil). *Cinnamomum tamala* oil also contains eugenol up to 75 to 90 percent and is a very potential source of eugenol.

0.5 Eugenol, along with oil of clove, finds application in the food industry as a spice. For a long time, eugenol constituted the main source of vanillin and is still considerably used for this purpose. It is also used in pharmaceutical industry. It is represented by the following structural formula:



EUGENOL

(Molecular Mass 164·21)

0.6 In the preparation of this standard considerable assistance has been derived from 'Givaudan Index (Ed 2), 1961', published by Givaudan-Delawanna Inc., New York.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for eugenol.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall be free from sediment, suspended matter and adulterants.

3.1.2 The material shall be examined for its colour, clarity, suspended matter and sediment as prescribed in IS : 326 (Part II)-1980‡.

3.2 Solubility — The material shall be clearly soluble in 2 volumes of (60 percent *v/v*) ethanol when tested as prescribed under **8** of IS : 326-1968§.

3.3 The material shall also be tested olfactorily and especially for by-notes as prescribed under **4** and **5** of IS : 2284-1963‖.

3.4 The material shall also comply with the requirements given in Table 1.

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*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials: Part II Preliminary examination of perfumery materials and samples (*second revision*).

§Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

‖Method for olfactory assessment of natural and synthetic perfumery materials.

TABLE 1 REQUIREMENTS FOR EUGENOL

(Clause 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Colour	Colourless to yellow	Cl 4 of IS : 326-1968*
ii)	Odour	Characteristic spicy odour of cloves	Cl 4 & 5 of IS : 2284-1963†
iii)	‡Relative density at 27°C/ 30°C	1·048 4 to 1·059 4	IS : 326 (Part III)-1980§
iv)	‖Refractive index at 27°C/ 30°C	1·536 8 to 1·539 0	Cl 7 of IS : 326-1968*
v)	Phenol content, calculated as eugenol ($C_{10}H_{12}O_2$), percent by mass, *Min*	98	IS : 326 (Part XII)-1980§
vi)	Gas chromatographic analysis:		
	Eugenol, percent by mass, *Min*	98	Appendix A
vii)	Freedom from hydrocarbons	To pass test	Appendix B

*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

†Method for olfactory assessment of natural and synthetic perfumery materials.

‡The correction factor for relative density for each degree Celsius change in temperature is 0·000 87.

§Methods of sampling and test for natural and synthetic perfumery materials:

Part III Relative density (*second revision*).
Part XII Determination of phenols (*second revision*).

‖The correction factor for refractive index for each degree Celsius change in temperature is 0·000 46.

4. PACKING AND MARKING

4.1 Packing — The material shall be supplied in glass or aluminium bottles, or any suitable container as agreed to between the purchaser and the supplier.

4.2 Marking — Each container so filled shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number and date of manufacture; and
d) Net and gross mass.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material, each sample containing not less than 50 ml shall be drawn as prescribed under 3 of IS:326-1968*.

5.2 Number of Tests

5.2.1 The phenol content shall be tested on each of the individual samples.

5.2.2 Test for determination of all the remaining characteristics given under 3 shall be tested on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples* — The test results on phenol content shall be recorded. Mean ($\bar{X}$) and range (R) of the test results shall be calculated as below:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum values of the test results

5.3.1.1 The lot shall be declared as conforming to the requirement for phenol content if the expression ($\bar{X} - 0{\cdot}6\,R$) is greater than or equal to 98.

5.3.2 For declaring the conformity of the lot to the requirements of all other characteristics determined on the composite sample, the test results for each of the characteristics, shall satisfy the relevant requirements given under 3.

*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

6. TEST METHODS

6.1 Tests shall be conducted as prescribed in IS : 326-1968*, IS : 2284-1963† and Appendices A and B. Reference to relevant parts and clauses of the standards and appendices is given in col 4 of Table 1.

6.2 Quality of Reagents — Unless specified otheswise, pure chemicals and distilled water (*see* IS : 1070-1977‡), shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (vi)]

GAS CHROMATOGRAPHIC ANALYSIS OF EUGENOL

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram using such a chromatograph is shown in Fig. 1 with the following chromatographic conditions:

Sample	Eugenol
Column	Carbowax 20M, 10 percent by mass in Chromosorb WAW 250-micron IS Sieve
Column temperature	220°C
Inject port temperature	200°C
Detector	Thermal conductivity of temperature 300°C
Carrier gas	Hydrogen with flow of 40 ml per minute
Chart speed	6·35 mm per minute
Attenuation	4

*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

†Method for olfactory assessment of natural and synthetic perfumery materials.

‡Specification for water for general laboratory use (*second revision*).

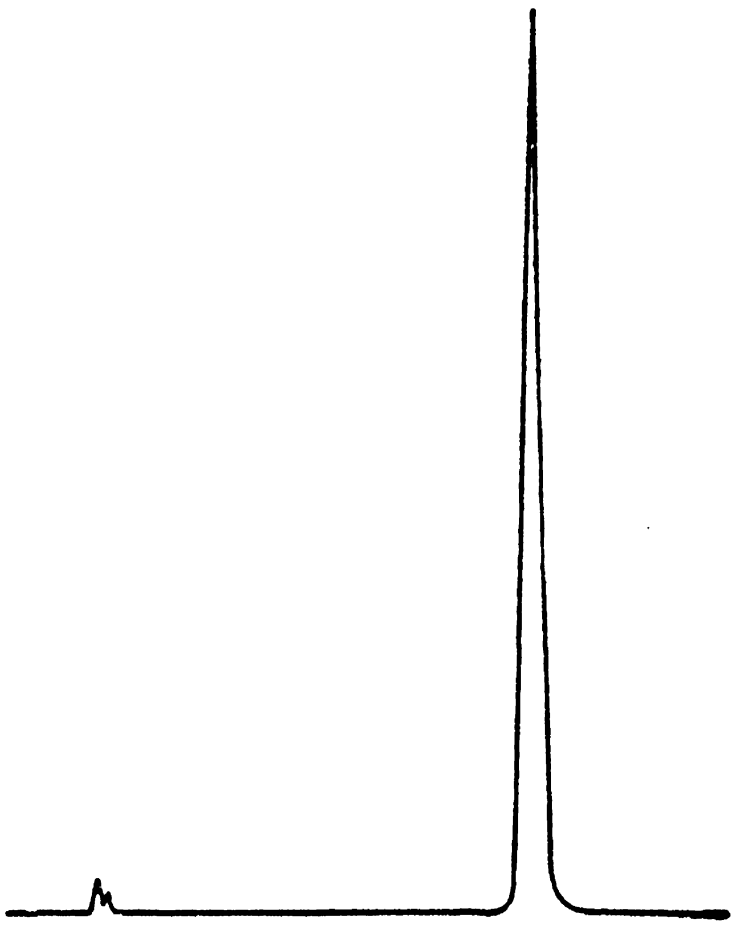

FIG. 1 TYPICAL CHROMATOGRAM OF EUGENOL

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject sample at injection port where it is vapourized and well mixed with carrier gas. This is led into the chromatographic column. The constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For this separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives the signal corresponding to the amount of particular constituents leaving the column. The detector signals, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-3. CALCULATION

A-3.1 Calculate, from the peak areas of individual constituents shown on the chromatogram of the material, concentrations of the constituents on the basis of peak areas on chromatograms obtained with known amount of pure constituents using the same apparatus under identical conditions.

APPENDIX B

[*Table* 1, *Item* (vii)]

DETERMINATION OF FREEDOM FROM HYDROCARBONS

B-0. GENERAL

B-0.1 Outline of the Method — Solubility of the material in sodium hydroxide solution is determined. A clear solution indicates the absence of hydrocarbons.

B-1. REAGENT

B-1.1 Sodium Hydroxide Solution — 0·5 N.

B-2. PROCEDURE

B-2.1 To 1 ml of the material contained in a 50-ml stoppered tube, add 20 ml of sodium hydroxide solution and 18 ml of water, and shake.

B-2.1.1 A clear mixture shall be immediately produced which may become turbid on exposure to air.

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